2nd Surface Mirror Cleaning and Verification

By

Dr. M. K. Barsh

Aerojet ElectroSystems Company
Azusa, California

The requirement that an advanced space satellite be free of contamination upon orbit insertion resulted in more stringent efforts to ensure maximum cleanliness at launch. Special techniques were developed to clean 2nd surface mirror thermal control radiator surfaces and ancillary procedures were invoked to verify their cleanliness level at launch.

Panels utilizing as many as 10,000 separate mirrors are used to achieve thermal control of the satellite system. Since the mirror panels can be exposed to a variety of potential contaminating environments during the satellite history of fabrication, assembly, ambient/vacuum testing, storage, shipment, and launch readiness, methods were studied to determine how best to clean and subsequently verify mirror cleanliness before launch. Whereas simple wiping of the surfaces with fabrics and appropriate solvents to achieve "visual clarity" may leave residues of as much as 1000 Å of surface contaminant, it was not evident how much improvement could be achieved by additional cleaning.

An effective cleaning procedure was established for 2nd surface-thermal-control mirrors using Auger Electron Spectroscopy to measure the contaminant residues on variously cleaned mirror panels. It was subsequently found that two sequential cleaning operations performed on individual mirror elements after the "visual clarity" condition had been obtained would achieve a minimal contamination level of less than 20 Å on the mirrors. In addition, a post-cleaning wipe-and-extraction procedure provided a means of verifying the level of cleanliness achieved. These techniques, currently employed to provide maximum 2nd surface-mirror cleanliness at launch of Aerojet space systems, will be described.

SURFACE CLEANLINESS

1.0 INTRODUCTION AND SUMMARY

The requirement that many of the newer more sophisticated satellite systems be relatively free of contamination for effective orbital performance demands that the cleanliness at launch be maximized. The thermal control surfaces of these satellite systems such as second surface mirrors are most critical with respect to the contamination levels. In order to effect initially "clean" critical surfaces, it was first necessary to evaluate the contamination levels of these surfaces, then to establish what levels of cleanliness could be achieved, and finally develop a practical procedure for obtaining and verifying the desired cleanliness level. Auger electron spectroscopy was utilized to determine the contamination levels on typical second surface mirror specimens and a cleaning and in-field verification procedure was developed which insured that the initial contamination levels on the mirrors would not be greater than approximately 20Å. The details are described below.

2.0 BACKGROUND

Satellite systems which require maintenance of low temperatures to insure optimum performance of critical subsystems often use passive radiators constituted of second surface mirrors whose ratio of absorptance to emissivity, α/ϵ is favorable for rejecting the maximum radiative energy while concomitantly absorbing the minimum. The presence of accumulated contamination on these mirrors increases their α and consequently degrades their thermal control characteristics. While it is evident that some contamination will collect on the systems during ascent and during orbital flight with time, it is obvious that

the lower the initial level of contamination at launch, the longer the system will be able to perform efficiently. The identification that accumulated contamination could be responsible for a degradation in system performance with time, led to one phase of a dedicated contamination program effort to insure that a minimum of contaminants initially resided on the mirrors at launch.

Initial activities consisted of cleaning to visual clarity, but it was recognized that this might leave as much as 1000 Å on the surfaces. When preliminary studies with the Fullam Laboratories Corp.[1] indicated that moisture condensation on solid mirror surfaces would occur preferentially where surface contamination was significant, a cleaning study was instituted to explore what cleaning procedure would be required to guarantee minimum contamination. The initial cleaning study by Fullam involved an initial scrubbing with "Milk of Magnesia," followed by water rinses and with final detergent solution scrubbing and final water rinses. The last step constituted a final wipe with Freon 113 and drying in a clean environment. Unfortunately, interpretation of the results was muddied because it was found that the condensation patterns were strongly influenced by the thermal conductance related to the mode of attachment of the mirrors to their supportive substrate. Although the study did demonstrate that multiple

1. Fullam, E.F. (Inc.), "The Attachment, Cleaning and Preservation of a Highly Reflective Surface," Report No. 1486 for Aerojet ElectroSystems Company, 9 Mar 1972.

cleanings, beyond visual clarity, could significantly improve the cleanliness level of the mirror surfaces, it was not evident how many recleanings were provident.

An alternate possibility of in-orbit cleaning by electrical discharge plasma was briefly investigated.[2] This required a bombardment of the mirror surfaces with energetic ions such as are found in an oxygen plasma. Whereas the laboratory results showed small but significant improvement by this technique, this approach was not pursued further because of the obvious difficulties in producing and maintaining a plasma over a large surface while in space.

Another approach to cleaning was considered. This consisted of applying collodion to the mirror surfaces, allowing the material to dry and then peeling the collodion away.[3] The procedure applied one or more times which requires a pure cellulose nitrate solution in ether-methanol mixture and a cheesecloth reinforcement, is purported to provide a very clean surface. Unfortunately attempts to apply this approach would wreak serious damage to the multimirror panels since the fluid collodion would migrate into the gap between the mirrors. Then, as the solidified collodion was peeled off to remove the contaminants, some of the individual mirrors might also be lifted off or damaged. Whereas the remaining mirrors might be reasonably clean, this would be offset by the extent of damage resulting from the peeling action. Consequently this approach was not pursued further.

2. Cruz, G.A. and Gillette, R.B., Active Cleaning Technique for Removing Contamination from Optical Surfaces in Space, The Boeing Co. Quarterly Progress Report No. 4 for GMSFC, Huntsville, Alabama, April 1, 1972 to June 1, 1972.

3. Tyndall, John B, Collodion Technique of Mirror Cleaning, NASA TECH BRIEF 70-10463, August 1970.

The primary problem in establishing the prerequisite cleaning procedure became one of determining quantitatively the resident contamination. A series of exploratory experiments with ellipsometry proved disappointing as it became evident that likely contaminants, such as silicone resin or adhesive could not be resolved effectively from the fused silica substrate because of the similarities in the indices of refraction. Ordinary optical techniques were rejected since the anticipated small thicknesses of contaminant (10-500 Å) precluded most of the techniques available.

A brief consideration of the constitution of probable contaminants revealed that the element carbon, in a wide variety of organic materials, would be a logical tracer of the presence of contaminants. Since the Auger Electron Spectroscopy technique provided a means of tracking elementary species down to very low levels, it was explored in depth. This technique, in conjunction with argon ion sputtering subsequently enabled quantitative estimation of residual contamination and permitted the establishment of a viable cleaning and verification technique. The details are discussed below.

2.1 Auger Electron Spectroscopy Studies

Preliminary studies were undertaken at Physical Electronics Inc. (PEI)[4] to evaluate the feasibility of quantitatively establishing the contamination thicknesses residing on cleaned second surface mirrors. Carbon, oxygen and silicon spectra were explored (Figure 1) and the peak-to-peak distances from the curve traces were used as a measure of the quantity of contaminant present when the contaminant levels were in the range of 50 Å. Further it was subsequently suggested by Dr. Palmberg (of PEI) that the C to Si peak-to-peak ratios would

4. Palmberg, H., Private Communication, Physical Electronics Industries, Inc., Edina, Minnesota, December 1972.

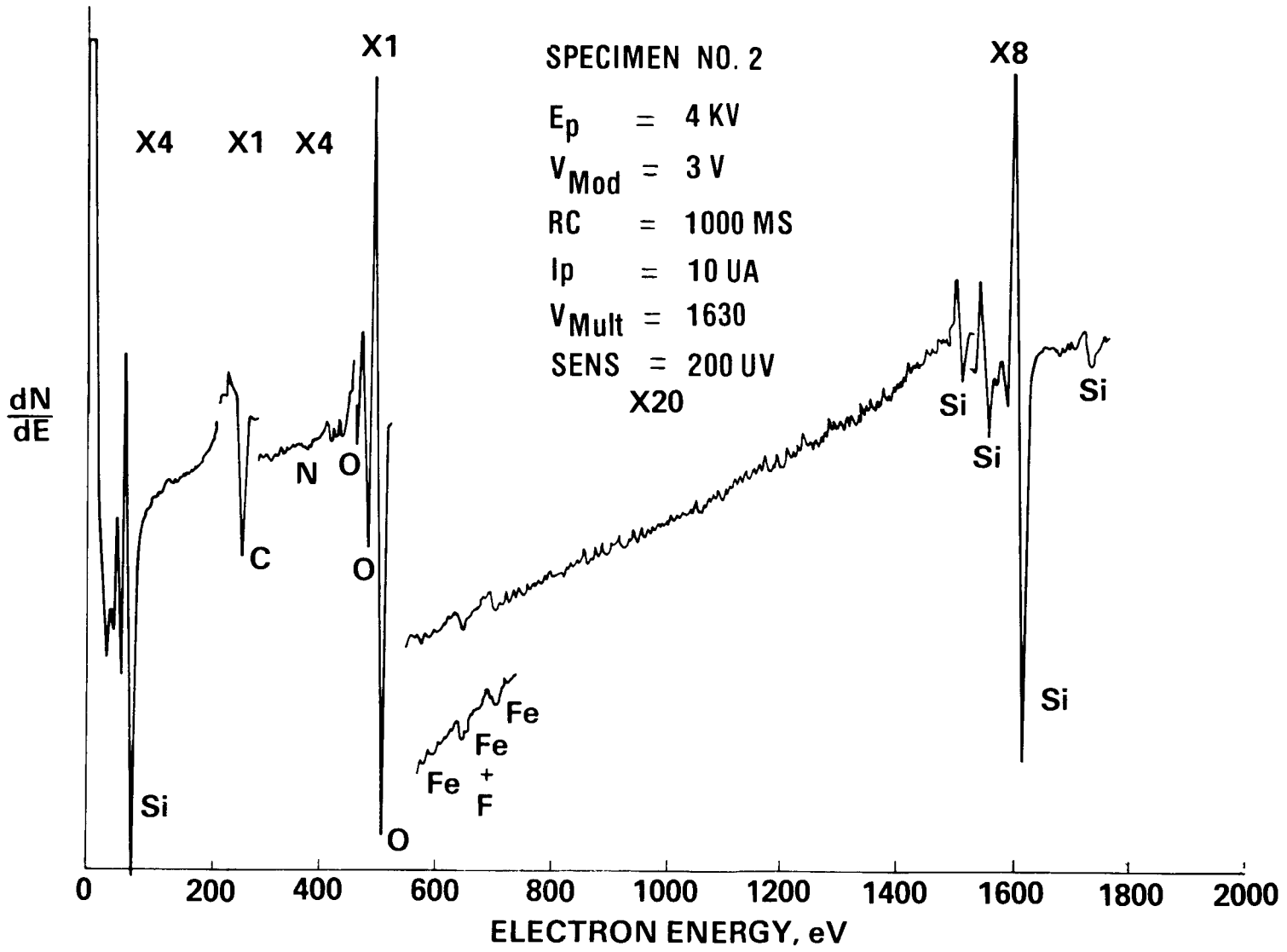

FIGURE 1 CONTAMINATION SPECTRA

provide a more accurate evaluation of the quantities of contaminant since the spectra of both would be evident during analysis. Further it was noted that the use of an argon ion sputtering beam could be used to etch away the contaminant while the Auger analysis was occurring so that a continuous record could be had of the contaminant removal with time. Using the etching technique it was relatively simple to establish when all of the contaminant was removed since the carbon peaks characteristic of the contaminants would disappear. Although silicon and oxygen peaks would vary somewhat during the sputtering, it was nevertheless not difficult to distinguish between the contaminant and substrate peaks.

A variant of the Argon sputtering and simultaneous Auger analysis was subsequently invoked which provided a relatively direct thickness calibration of the carbon-to-silicon peak ratios. Extensive exploration of a wide variety of organic materials had revealed that a sputtering beam of a given energy would etch away the organic material at a known fixed rate. The equipment was then programed to repetitively scan over a very narrow band in a sawtooth mode corresponding to the carbon peak. With the recording graph paper driven at constant speed, and the AES activated, the sputtering beam was turned on and maintained until the carbon peak-to-peak values had dwindled to zero (Figure 2). It was then a simple matter to estimate the initial thickness of contaminant and to correlate it with the C to Si ratios.

2.2 Cleaning Studies

The use of the thickness measuring tool described above enabled a cleaning evaluation study to be undertaken. This consisted of evaluating several cleaning materials and techniques. In order to fully appreciate the problem it is enlightening to consider the way in which the second-surface-mirror temperature control (TC) surfaces were constituted. The TC panels consisted of

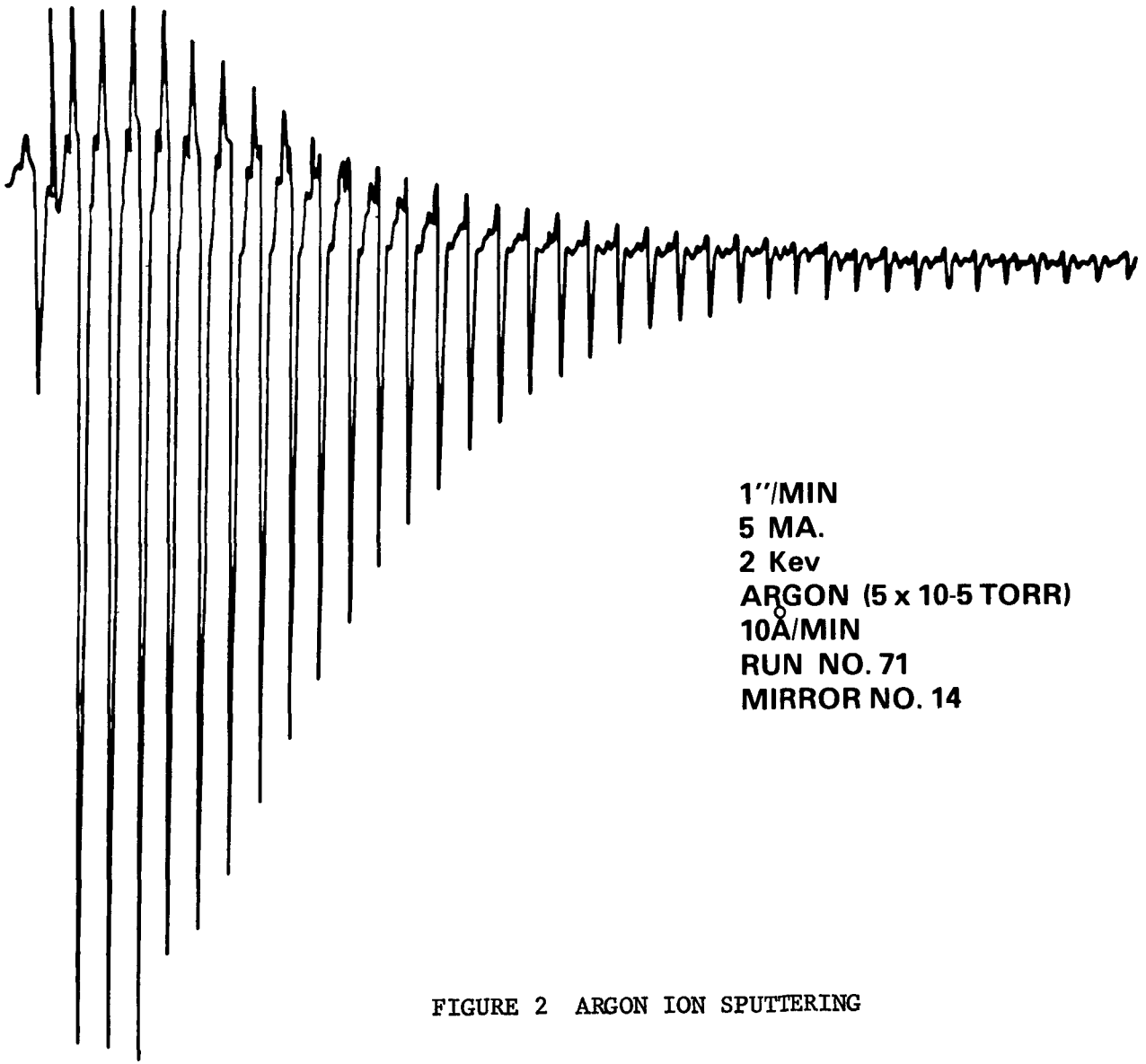

FIGURE 2 ARGON ION SPUTTERING

one inch square second-surface fused silica mirrors bonded to a magnesium substrate. However, in order to accommodate thermal excursions and accompanying expansion and contraction forces, the mirror panels, as fabricated, were separated by a gap of 0.006 inches. In order to clean these panels, account had to be taken of the problem of "wiping" contaminants into the gaps and subsequently extracting the contaminant out to spread over the mirror surfaces. Conceptually, the cleaning procedure was divided into two phases: (a) general overall wiping with appropriate cloths and solvent and (b) specific individual mirror cleanings to circumvent the gap problem. The questions which required resolution were: (1) how much initial/general wiping should be done and to what criteria; (2) what type of wiping material; (3) what solvents; (4) how should the individual mirrors be cleaned; (5) what type of cleaning materials; (6) what solvents; (7) how many repetitive cleanings are necessary to achieve minimum cleanliness; and (8) how could this level of cleanliness be verified both at the factory and at the launch site.

The first problem that was approached was to select a method and the materials for the cleaning. Since, at the inception of this effort, no major modification of the space system was considered acceptable, it was necessary to choose a technique that would be practical for both the factory and launch sites. The method selected was that implied above; namely, that an initial overall wiping to visual clarity would be followed by repetitive individual mirror recleaning wipings. Several materials and solvents were considered and were tried on individual mirrors of the mirror panels which had been artificially dirted with RTV566, a typical tenacious contaminant candidate. The mirrors were subsequently analyzed by the Auger technique and the results are shown in Figure 3. It may be seen that the smallest

SOLVENT SYSTEM	DEPOSITS REMAINING
Detergent*	> 50 Å
Detergent/Perchloroethane*	> 8 Å
Ethanol/90-10, 1,1,1 Trichloroethane, Ethanol**	6.5 Å

*Process studied by Aerospace Corporation

**Solvent mixture contains less than 1 ppm of non-volatile residue.

FIGURE 3 EFFECTS OF SOLVENT ON CLEANING EFFECTIVENESS

deposit was obtained using the 1,1,1 trichlorethane/ethanol solvent mixture. The cloths selected for the wipings, optic cotton cloth, were those which would not degrade with 150 cycle Sohxlet extractions with methanol (found to be most effective) and would be reasonably wetted by the selected solvents.

The next problem approached was the maximum practical level of cleanliness that could be achieved assuming the use of a general precleaning followed by repetitive individual mirror cleanings. Clearly an infinite number of repetitive cleanings appear superficially to be ideal but it was anticipated that something less than this would prove to be adequate. The approach taken was to fabricate a series of 2 by 2 mirror panels (4 mirrors total) incorporating the expansion gaps described above (see Figure 4). These panels were limited to this size to accommodate their introduction into the Auger equipment. The panels were then variously "dirtied." Some were handled with incipient contamination whereas others were carefully and "excessively" precleaned and subsequently "redirtied" with RTV566 adhesive. The mirror panels were treated in various ways prior to insertion into the Auger system. They were all initially subjected to a general overall wipe-cleaning to visual clarity; i.e., no evidence of any contamination could be seen with the naked eye when illuminated by the use of a narrow collimated light beam. Then individual mirrors were carefully cleaned, some once, others twice, and more. Auger measurements were subsequently made on the panels and the results are summarized in Figure 5. It can be seen that no significant improvement is to be expected if the individual mirrors are cleaned more than 2 times each, after visual clarity had been achieved and that the minimum contaminant thickness would be about 8-10 Å.

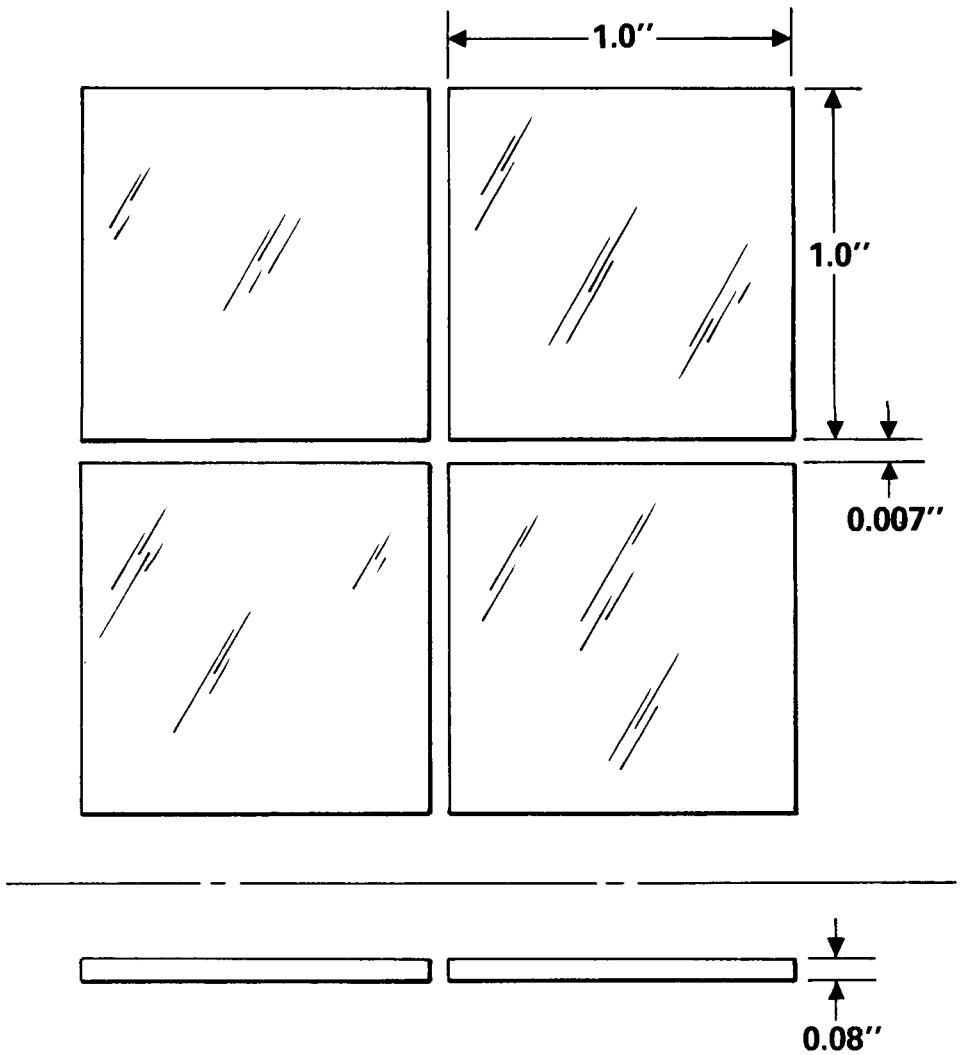

FIGURE 4 MIRROR TEST PANEL

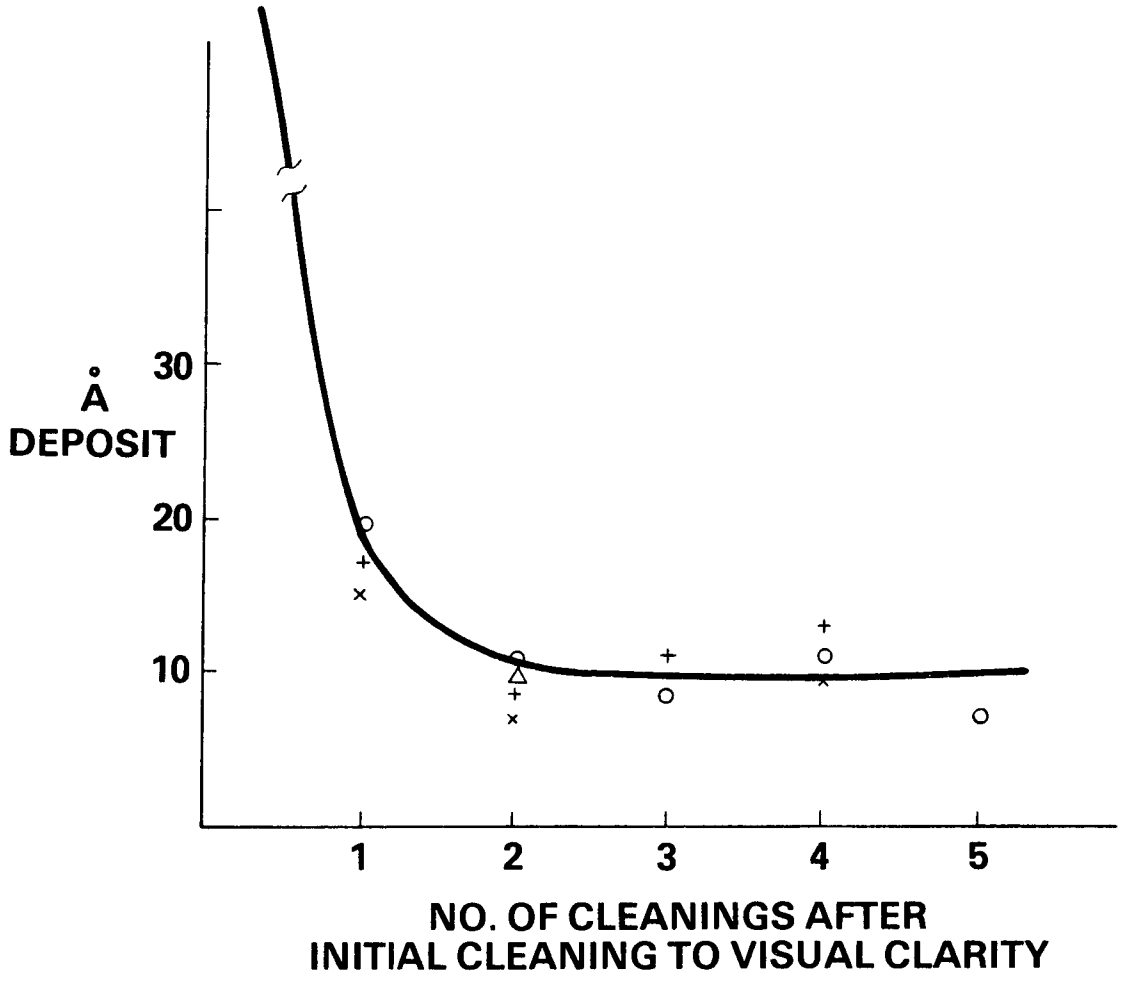

FIGURE 5 EFFECTS OF MULTIPLE CLEANINGS

The remaining problem, the development of a practical cleanliness verification technique, was solved using a variation of the cleaning procedures described above. A set of approximately 300 mirrors in a typically assembled panel were individually cleaned using one small piece of cloth for two mirrors after first having been cleaned overall to visual clarity. Another 300 pieces of extracted cloth were set aside for the second individual mirror cleaning. 150 pieces were used for cleaning the 300 mirrors and were subsequently stored in a clean bottle. The remaining 150 cloth pieces were handled in the same way as the cleaning cloths (including wetting with solvent) except that instead of being used to wipe mirrors, they were placed one-at-a-time into another clean bottle at the same time that the used mirror-cleaning-cloths were being placed in their bottle. The intent of this duplicate procedure was to insure that the non-cleaning cloths would experience the same environment except for the mirror cleaning and therefore could provide a valid blank as a reference. Both sets of cloths were then Sohxlet extracted for 90 cycles (minimum) with chloroform and the solvent carefully evaporated to dryness. The non-volatile residues (NVR) were weighed and the non-cleaning cloths data provided a tare for correcting the cleaning cloths data. The results of a typical example are summarized in Figure 6. The residue are primarily organic and are assumed to have a density of approximately 1 gm/cm^3. Thus it follows that the net amount extracted, 0.0002 gm, is equivalent to about 10 Å depth covering the area of 300 one inch mirrors. In view of the minimum thickness achievable as indicated in the previous paragraph, this implies that the contaminant was partitioned equally between the cleaning cloths and the mirror surfaces, and the total contamination on the mirrors, prior to the 300 mirror verification cleaning must have been $\sim$ 20 Å. Since it was demonstrated previously that more than 2 sequential cleanings did not achieve greater cleanliness, this value of 0.0002 gm extracted from 300 mirrors can be used as a criterion for establishing a practical maximum cleanliness level. This, after mirror panels were

Sample	NVR Wt. (mg.)
• 150 cloths from cleaning 300 1-in. mirrors	0.57
• Tare from 150 cloths	0.37
• Net contaminant	0.20 mg.

FIGURE 6 VERIFICATION DATA

believed to have been adequately cleaned to visual clarity, a sample consisting of a 300 mirror panel was recleaned, one mirror at a time, with 150 pieces pre-extracted cloth and pure solvent mixture. The requirement was subsequently stipulated that the net extract from the cloths used for the 300 mirror cleaning shall be equal or less than 0.0002 gm to insure that the contamination on the mirror panels would be equal or less than 20 Å.

3.0 CONCLUSIONS AND RECOMMENDATIONS

The validity of the extractive approach is predicated on the assumption that all contaminants would be partitioned between the mirror surfaces and the cloths in the same manner as it was in the test procedure. For ordinary contaminants derived from handling or even from the possible deposition or surface migration of tenacious materials, such as the silicones, this is probably true. However, it is obvious that a fully cured epoxy resin on the mirror surface might not provide valid data in the cleaning-extraction process. Perhaps the crux of the matter lies in the fact that the first step in the cleaning procedure requires "visual" clarity; a condition which could hardly be obtained with a fully cured epoxy. Further, additional tests using mirrors predirtied with various silicone adhesives and oils, epoxy, acrylic and polyurethane adhesives gave essentially the same results once the condition was met that the initial visual clarity cleaning was achieved.

One of the concerns with the one-at-a-time mirror cleaning technique was the possibility that with the cleaning of large numbers of mirrors using extreme care to avoid crossing over the gaps between the mirrors, some of the mirrors might not be cleaned to their extremities as a result of fatigue, unintentional carelessness, etc. The resultant effects might not be picked up by the verification technique since it was basically the same as the cleaning technique.

Subsequent experiments with multiple overall wipes with larger pieces of cloth suggest that a 10-fold repetitive procedure might accomplish the same as one overall wipe to visual clarity followed by 2 sequential individual mirror cleanings. The 10-fold procedure appears more attractive than the separate mirror especially where 10,000 or more separate mirrors are involved. This alternate procedure is still under investigation as it has not yet been demonstrated that it might not wipe the surface contaminants into the gaps. Subsequently the contaminants might migrate out onto the mirror surfaces to defeat the intention.

With reference to verification techniques, the state-of-the-art of the configuration of spacecraft has developed recently to such an extent that it now appears attractive and feasible to incorporate small witness plates of 4 individual mirrors each into the second surface mirror thermal control surfaces. These witness plates could subsequently be removed to an Auger Spectrometer and their surfaces directly analyzed for contamination. This would circumvent the obvious shortcomings of the extractive verification procedure. This alternate approach appears most attractive and is currently under consideration for use with some of the space systems fabricated by Aerojet.

4.0 REFERENCES

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2. Cruz, G.A. and Gillette, R.B., Active Cleaning Technique for Removing Contamination from Optical Surfaces in Space, The Boeing Co. Quarterly Progress Report No. 4 for GMSFC, Huntsville, Alabama, April 1, 1972 to June 1, 1972.
3. Tyndall, John B, Collodion Technique of Mirror Cleaning, NASA TECH BRIEF 70-10463, August 1970.

4. Palmberg, H., Private Communication, Physical Electronics Industries, Inc., Edina, Minnesota, December 1972.